Pitfalls in borate fusion: working with calcinated samples

Abstract

Here, we demonstrate the significance of crucible material for calcination of CRMs or QC samples. Two sets of NIST CRM 1880b – 1889b samples of Portland cement were calcinated either in ceramic or in platinum crucibles. The calcinated material was fused using Lithiumtetraborate in a Bead One HF and measured in a Bruker-AXS S8 TIGER II. The data shows contamination of samples with K_2O and Na_2O which were calcinated in the ceramic crucibles. Inevitably, the samples reacted with the ceramic crucible at only 950 °C leading to irregular contamination with the alkali metals. In platinum crucibles, the Portland cement CRM could be prepared without any contamination.

Key words

• **Borate Fusion** • **Contamination** • **Bead One HF** • **Bead One R** • **HAG-HF** • **XRF**

Introduction

Certified reference material (CRM) can be used to calibrate e.g. XRF spectrometers. Using the known elemental concentrations, the XRF spectrometer calculates a calibration line with the measured intensities of prepared samples. Grinding duration, pressing force or fusion duration and fusion temperature must be strictly kept constant for calibration and quality control samples. However, there might be other pitfalls which can deteriorate the calibration of the XRF spectrometer by using incorrect crucibles for LOI detection.

Often, samples are calcinated at 950 – 1050 °C in order to remove carbonates, crystal water or to oxidize reducing phases which could severely damage platinum crucibles during fusion. During this process, samples might react with the crucible, eventually contaminating the sample.

Methods

We used $Pt_{95}Au_5$ and glazed porcelain melting crucibles for this test. The crucibles were filled with 5 g NIST Portland cement CRM and calcinated at 950 °C until constant weight. Subsequently, we fused 0.6 g of the

calcinated material using 6 g Lithiumtetraborate in order to form 32 mm glass beads. A Bead One HF induction based fusion system was used for the sample preparation (Figure 1).

Figure 1: *Bead One HF used for the fusion of the material.*

We used temperature calibrated crucibles (Application Notes 23 & 24) for increased repeatability (Figure 2). Automatic fusion was done in $Pt_{95}Au_5$ crucibles for 10 minutes at 980 °C.

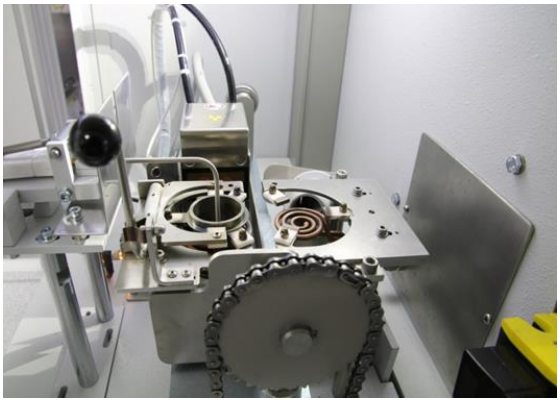

Figure 2: *Calibration unit (Application Note 23 & 24) used to increase repeatability.*

Table 1 shows the CRM set used for this test. Of each CRM, we prepared duplicates to give an indicator about precision.

Table 1: *Certified reference material of NIST that was used for the calibration.*

NIST CRM			
1880b	1881b	1884b	1885b
1886b	1887b	1888b	1889b

A Bruker-AXS S8 TIGER II 4kW sequential wavelength-dispersive X-ray spectrometer with a 75 positions automatic sample changer with easy Loader and a rhodium end-window X-ray tube was used for data generation. 40 kV and 100 mA was used for the measurement. Total measurement time for 13 elements was 200 s. A 28 mm collimator mask and vacuum were used for all measurements.

Results

Figure 3 and Figure 4 show the calibration results of K and Na respectively. The coefficient of determination is higher for samples prepared in platinum crucibles. For K, R^2 is 0.999 for CRMs prepared in platinum crucibles while CRMs prepared in ceramic crucibles only have R^2 = 0.9837. Likewise, the same relation is true for Na. However, R^2 only decreased to 0.9982 from 0.9992 indicating either less or more constant contamination from the crucible.

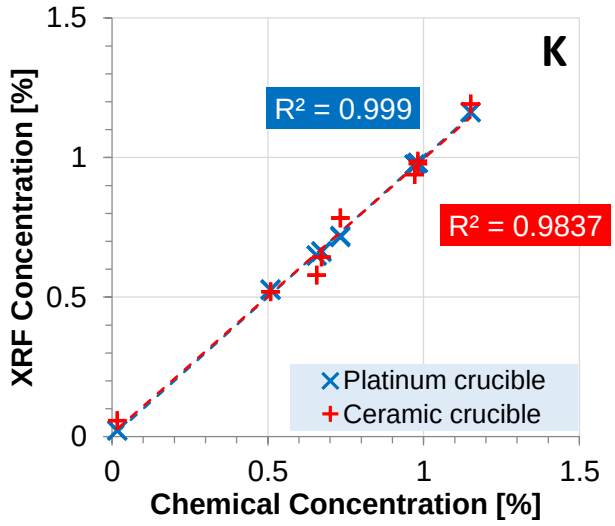

Figure 3: *Calibration results obtained for Potassium.*

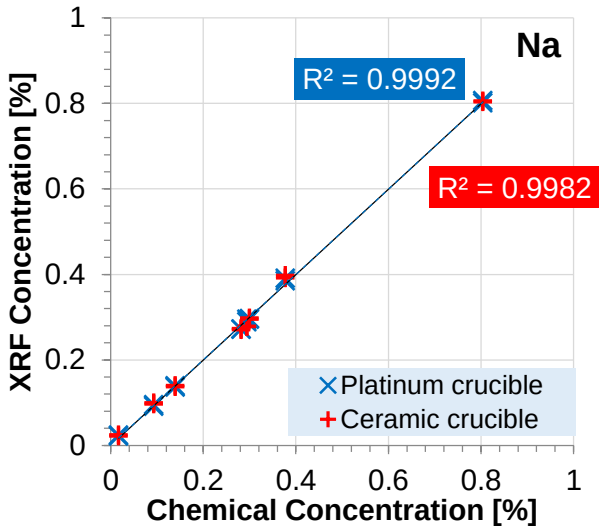

Figure 4: *Calibration results obtained for Sodium.*

Discussion

The preparation of CRM samples for calibration with prior calcination can be critical depending on the crucible material used. During calcination, samples might react with ceramic crucibles and contamination can occur. The contamination with K and Na very likely comes from feldspars used in the manufacturing of the glazed porcelain melting crucibles. Especially K showed significant contamination which decreased R^2 from 0.999 to 0.9837. Although both linear regressions are almost identical, ceramic crucibles should be avoided for samples which are supposed to be analyzed.

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